

CLAIMS

1. Process for the preparation of particles comprising at least one metal ion which comprises the following stages:

- 5 a) at least one precursor comprising a metal cation is dissolved or dispersed in an aqueous medium;
- b) a partial hydrolysis of said precursor is optionally carried out,
- c) the precursor resulting from stage a) or the
- 10 partially hydrolyzed precursor resulting from stage b) is brought into contact with at least one water-soluble comb copolymer comprising either a complexing anionic backbone and stabilizing hydrophilic side chains or a stabilizing hydrophilic neutral backbone and complexing
- 15 anionic side chains or at least one of the two abovementioned copolymers in combination with at least one complexing anionic hydrophilic polymer;
- d) a partial or complete hydrolysis of the product obtained during stage c) is carried out.

20 2. Process according to Claim 1, characterized in that the metal cation is chosen from the metals from Columns IIIA, IVA, VIII, IB, IIB, IIIB and VB of the Periodic Table, the lanthanides and the actinides.

25 3. Process according to Claim 2, characterized in that the metal cation is chosen from titanium, iron, cobalt, nickel, copper, aluminum, zinc,

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gold, silver, platinum, cerium, lanthanum, yttrium, iridium, ruthenium, rhodium, osmium, palladium or their mixtures.

4. Process according to one of the preceding claims, characterized in that the precursor is in the form of an aqueous solution of a water-soluble salt of a metal cation chosen from nitrates, sulfates, chlorides, phosphates or their mixtures.

5. Process according to one of Claims 1 to 4, characterized in that the precursor is in the form of an aqueous dispersion of particles or of aggregates of particles comprising a hydroxide, a hydroxide oxide or a partially hydrolyzed water-soluble salt of a metal cation, alone or as mixtures, optionally combined with an oxide of a metal cation.

6. Process according to the preceding claim, characterized in that the particles or the aggregates have a mean size of less than or equal to 100 nm, preferably of between 2 and 100 nm.

7. Process according to one of the preceding claims, characterized in that the hydrolyses of stage b) and that of stage d) are carried out in the presence of a base chosen from alkali metal or alkaline earth metal hydroxides and aqueous ammonia.

8. Process according to Claim 7, characterized in that the base is chosen from sodium

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hydroxide, potassium hydroxide, calcium hydroxide or aqueous ammonia, alone or as mixtures.

9. Process according to one of the preceding claims, characterized in that the amount of  
5 base employed during stage b), if it takes place, and during stage d) corresponds to 50 to 130% of the stoichiometric amount needed to completely hydrolyze the precursor.

10. Process according to the preceding  
10 claim, characterized in that, if  $n_1$  is non zero and represents the number of moles of base employed during stage b),  $n_2$  represents the number of moles of base employed during stage d) and  $n$  represents the sum of  $n_1$  and  $n_2$ , then  $n_1$  and  $n_2$  conform to the following  
15 inequalities  $0 < n_1 \leq 0.8n$  and  $0.2n \leq n_2 < n$ .

11. Process according to one of the preceding claims, characterized in that the water-soluble comb copolymer, optionally combined with the water-soluble polymer, is chosen so that the comb  
20 copolymer, optionally combined with the hydrophilic polymer, forms a transparent solution at 10% by weight in water at the lowest temperature to which said comb copolymer, optionally combined with the hydrophilic polymer, is subjected in the process.

25 12. Process according to the preceding claim, characterized in that the weight-average

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molecular mass (Mw) is between 2 000 and  $5 \times 10^5$  g/mol, preferably between 3 000 and  $10^5$  g/mol.

13. Process according to either of Claims 11 and 12, characterized in that the water-soluble comb  
5 copolymer comprises a complexing anionic hydrophilic backbone and nonionic stabilizing hydrophilic side chains, said backbone being obtained from monomers chosen from unsaturated monocarboxylic acids, unsaturated polycarboxylic acids or their anhydride  
10 form, or unsaturated sulfonic acids, optionally in combination with one or more water-insoluble monomers.

14. Process according to Claim 13, characterized in that the monomers forming the nonionic side chains are macromonomer entities chosen from  
15 macromonomers of poly(ethylene glycol) (meth)acrylate, poly(vinyl alcohol) (meth)acrylate, poly(hydroxy(C<sub>1</sub>-C<sub>4</sub>)-alkyl (meth)acrylate) (meth)acrylate, poly(N-methylol-acrylamide) (meth)acrylate or poly((meth)acrylamide) (meth)acrylate type.

20 15. Process according to the preceding claim, characterized in that the nonionic side chains exhibit a poly(ethylene glycol) number-average molar mass of between 200 and 10 000 g/mol, preferably between 300 and 2 000 g/mol.

25 16. Process according to either of Claims 11 to 12, characterized in that the copolymer comprises a stabilizing hydrophilic neutral backbone and complexing

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anionic hydrophilic side chains, said neutral backbone being obtained from ethylene oxide in the form of an oligomer or of a polymer.

17. Process according to the preceding  
5 claim, characterized in that the side chains are obtained from monomers chosen from unsaturated carboxylic acids, polycarboxylic acids or their anhydride form, unsaturated amino acids or unsaturated sulfonic acids.

10 18. Process according to any one of Claims 11 to 17, characterized in that the monomers forming the complexing anionic backbone or the complexing anionic side chains can be combined with, or partially substituted by, esters of unsaturated carboxylic acids,  
15 optionally carrying a sulfonated group or a hydroxyl group; esters of unsaturated carboxylic acid; linear or branched hydrocarbonaceous monomers comprising at least one carbon-carbon double bond which comprise 2 to 10 carbon atoms in the longest chain; vinylaromatic  
20 monomers;  $\alpha,\beta$ -ethylenically unsaturated nitriles;  $\alpha,\beta$ -ethylenically unsaturated amides; vinyl ether; or N-vinylpyrrolidone.

19. Process according to one of Claims 11 to 18, characterized in that the copolymer is combined  
25 with at least one polymer obtained by polymerization of at least one anionic monomer chosen from unsaturated

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carboxylic acids, polycarboxylic acids or their anhydride form, or unsaturated sulfonic acids.

20. Process according to the preceding claim, characterized in that the weight-average molar mass of the polymer is between 2 000 and  $5 \times 10^5$  g/mol, preferably between 3 000 and  $10^5$  g/mol.

21. Process according to any one of the preceding claims, characterized in that the level of copolymer employed during stage c), which is the molar ratio of the complexing group of the copolymer of the anionic hydrophilic part or parts to the number of mole of the metal cation present in the precursor, is between 0.05 and 2, more particularly between 0.1 and 0.5.

22. Process according to one of the preceding claims, characterized in that the mean size of at least 80% of the particles obtained at the end of stage d) is between 2 and 500 nm, preferably between 2 and 300 nm.

23. Process according to any one of the preceding claims, characterized in that, after stage d), a stage e) of maturing is carried out at a temperature of between  $10^\circ\text{C}$  and a temperature of less than or equal to the boiling point of said dispersion.

24. Process according to Claim 23, characterized in that, after stage d) or after

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stage e), a stage f) a concentration of the dispersion is carried out.

25. Process according to Claim 24, characterized in that the concentration is carried out  
5 by partially or completely separating the particles from the medium of the dispersion and then optionally by redispersing the particles thus obtained in an appropriate amount of aqueous medium.

26. Process according to either of Claims 24  
10 and 25, characterized in that the separation stage can be carried out is by ultrafiltration, dialysis, precipitation, centrifugation or ultracentrifugation, by complete or partial evaporation, with or without heating, of the aqueous medium of the dispersion, or by  
15 lyophilization, it being possible for these stages to be carried out alone or in combination.

27. Particles capable of being obtained according to one of the preceding claims, characterized in that the mean size of said particles is between 2  
20 and 500 nm and preferably between 2 and 300 nm.

28. Use of the particles according to the preceding claim or capable of being obtained by the process according to one of Claims 1 to 26 in the mechanical polishing of hard objects, in the  
25 preparation of pigments or mixed ceramics for the electronics industry, in the reinforcing of polymeric matrices, in fungicidal or biocidal dispersions, in the

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scavenging of sulfur derivatives or in the scavenging  
of unpleasant smells.

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